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# CHRYSOTILE ASBESTOS DILUTION STUDIES

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# Chrysotile Asbestos Dilution Studies

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Report #66

A Joint Project of the Ministry of the Environment, Ontario and the Canada Centre for Inland Waters, Department of Fisheries and Environment, Canada

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## ABSTRACT

Laboratory studies involving the simple dilution of concentrated asbestos suspensions in various distilled water - river water mixtures produced erratic results in terms of the anticipated asbestos counts. Peptization of the water prior to the introduction of the asbestos suspension produced more accurate asbestos fibre counts than those encountered in the unpeptized dilutions.



## RÉSUMÉ

Des études en laboratoire ont révélé que la simple dilution de particules concentrées d'amiante en suspension dans divers mélanges d'eau douce et d'eau distillée, ne permet que des prévisions inégales du dénombrement des particules d'amiante. L'adjonction de peptides avant l'introduction des fibres d'amiante en suspension, a conduit à des dénombrements plus exacts de ces fibres.

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## INTRODUCTION

Pilot plant studies incorporating conventional potable water pre-treatment and filtration were carried out in 1975 in order to assess chrysotile asbestiform fibre removal (1). The Humber River raw water supply for the laboratory based pilot plant did not contain sufficient asbestos to make removal studies feasible and hence a 50 mg $\ell^{-1}$ "asbestos suspension" that had previously been developed (2) was metered into the raw water. Consistent fortification of the Humber River water was found not to be possible. After many modifications to the feeding system in order to optimize the mechanics of the feed system, a series of tests was carried out, the conclusion of which was that the asbestos particles appeared to enter the asbestos contact chamber with the raw water, but did not appear to leave.

Hence, a further set of experiments was carried out in order to determine if the apparent "disappearance" of the positively charged chrysotile asbestos fibres was due to the interaction with the negatively charged colloidal and particulate matter present in the river water.

The electrostatic behavior of the chrysotile particles was investigated further by a series of experiments in which asbestos suspension was added to dilution water that had been dosed with selected cationic polyelectrolytes. The polyelectrolyte addition would serve to stabilize the negatively charged particulate material in suspension by coating such particles with strong positive charges and thus resisting agglomeration with the positively charged asbestos fibres and hence effecting peptization.

## METHODS

Jar-test dilutions were made of the "asbestos suspension" with various ratios of distilled and river water. The asbestos suspension ($4.6 \times 10^{11}$ fibres $\ell^{-1}$) was diluted 1:10,000 and 1:1000 with various compositions

of water ranging from 100% distilled water through distilled water/river water mixtures to 100% river water and also with Toronto tap water. Dilutions were made in ultrasonically cleaned 1 litre plastic beakers, and the dilute suspensions were stirred at 100 rpm for approximately 3 hours before being sent for analysis.

To minimize the effect of the negatively charged particulate matter still further, the diluting water was dosed with a cationic polyelectrolyte prior to the addition of the asbestos suspension in order to effect peptization.

The polyelectrolytes and dosage selected for use were those which significantly increased the zeta potential of the asbestos suspension.

Three separate series of 1:10,000 dilutions were made of the asbestos suspension in distilled water with various doses of Cat-Floc B or Purifloc C31 (3). Samples of diluting water were treated with the polymer and stirred for 5 minutes to complete the peptization prior to the addition of the asbestos aliquot.

The asbestos counting for this project was carried out exclusively at the Canada Centre for Inland Waters. The counting was considered to be as precise as was possible considering the generally nebulous state-of-the-art. The reader should refer to the Appendix for further discussion of the analytical precision.

## RESULTS & DISCUSSION

### River Water - Distilled Water Dilutions

Table 1 shows the composition of the water used for the dilutions and the resulting fibre concentrations. In

Table 1

Jar-Test Dilutions of an Asbestos Suspension Containing $4.6 \times 10^{11}$ Fibres $\ell^{-1}$

| Sample # | % Distilled Water | % River Water | Turbidity (Ftu) | Asbestos Results (Fibres $\ell^{-1}$) |
|---|---|---|---|---|
| 1:10,000 dilution (calculated concentration $4.6 \times 10^7$ fibres $\ell^{-1}$) | | | | |
| 1 | 0 | 100 | 35 | N.A.* |
| 2 | 25 | 75 | 25 | $2.9 \times 10^6$ |
| 3 | 50 | 50 | 18 | $4.7 \times 10^6$ |
| 4 | 75 | 25 | 8 | $3.9 \times 10^6$ |
| 5 | 100 | 0 | 0.3 | $3.7 \times 10^6$ |
| 6 | Toronto Tap Water | | 0.4 | $1.0 \times 10^7$ |
| 1:1,000 dilution (calculated concentration $4.6 \times 10^8$ fibres $\ell^{-1}$) | | | | |
| 1 | 100 | 0 | 35 | $3.9 \times 10^6$ |
| 2 | 75 | 25 | 24 | N.A.* |
| 3 | 50 | 50 | 17 | $1.5 \times 10^6$ |
| 4 | 25 | 75 | 8 | N.A.* |
| 5 | 0 | 100 | 0.1 | $1.2 \times 10^6$ |
| 6 | Tap Water | | 0.1 | $6.1 \times 10^6$ |
| 1:1,000 dilution (triplicate repeats of above experiment for 100% distilled water and 50:50 distilled:river water) | | | | |
| D1 | 100 | 0 | 0.1 | $1 \times 10^7$ |
| D2 | 100 | 0 | 0.1 | $1 \times 10^8$ |
| D3 | 100 | 0 | 0.1 | N.A.* |
| R1 | 50 | 50 | 15 | $1 \times 10^7$ |
| R2 | 50 | 50 | 15 | $1 \times 10^8$ |
| R3 | 50 | 50 | 15 | N.A.* |

* N.A. - asbestos counts not available - dilutions were made but counts not carried out

the first set of data (1:10,000 dilutions), all the results are low by a factor of 3-4 except for the tap water dilution which agrees closely with the calculated concentrations. However, in the second set (1:1000 dilution), only the result of the 100% distilled water dilution agrees with the calculated concentration and all the other results are low by factors between 25 and 66. Obviously, there is no consistent trend in these results. The third set of results in Table 1 (triplicate 1:1000 dilutions in 100% distilled water and 50:50 distilled river water) clearly illustrates the inconsistent results obtained: for identical simple dilutions, the final concentrations vary by a factor of 10. From these results, it is impossible to determine whether it is the dilution process itself which is non-reproducible or inherent inaccuracies of the analytical procedure (either in the sample preparation or the actual counting of the filters).

Peptization in Dilutions

A separate series of experiments indicated that the addition of 1-3 mg $\ell^{-1}$ Cat-Floc B or Purifloc C31 to the concentrated asbestos suspensions significantly increased the zeta potential of the suspension.

The results of the jar tests in which Cat-Floc B or Purifloc C31 was added to the dilution water prior to the addition of the asbestos suspension are given in Table 2. The calculated concentration after dilution was approximately $4.6 \times 10^7$ fibres $\ell^{-1}$. It is apparent from the results that while the diluted suspensions containing the polyelectrolyte all contain in the order of $10^7$ fibres $\ell^{-1}$, those without the polyelectrolyte are much more unpredictable and range over two orders of magnitude. The data suggested that the apparent loss of fibres on dilution is in some way associated with neutralization in surface charge, but

Table 2

Effect of Polyelectrolytes on Jar-Test Dilutions of Asbestos Suspension Containing $4.6 \times 10^{11}$ Fibres $\ell^{-1}$

All dilutions 1:10,000 with distilled water

| Polyelectrolyte | | Asbestos |
|---|---|---|
| Type | Conc. mg $\ell^{-1}$ | Concentrations (fibres $\ell^{-1}$) |
| None | - | $1.3 \times 10^7$ |
| None | - | $7.0 \times 10^6$ |
| None | - | $1.0 \times 10^5$ |
| None | - | $2.4 \times 10^7$ |
| Purifloc C31 | 2.0 | $6.1 \times 10^7$ |
| Purifloc C31 | 1.0 | $6.2 \times 10^7$ |
| Purifloc C31 | 1.0 | $1.5 \times 10^7$ |
| Cat-Floc B | 2.0 | $3.6 \times 10^7$ |
| Cat-Floc B | 1.0 | $1.6 \times 10^7$ |
| Cat-Floc B | 1.0 | $2.7 \times 10^7$ |

on the other hand, one would then have predicted dilutions with river water to result in lower concentrations of fibres than corresponding dilutions with distilled water (due to the high ionic and suspended solids content of the former). Clearly, from Table 2, this is not the case. However, the addition of polyelectrolytes during dilution does seem to lead to more consistent results. The addition of polyelectrolytes may also prove a useful stabilizing technique for analytical samples when prolonged storage is expected.

## CONCLUSIONS

1. Consistently accurate quantitative analysis of chrysotile asbestos particles in water following simple dilution of a concentrated chrysotile asbestos suspension was not found to be possible. Generally, the counts obtained after dilution ranged from the anticipated concentration to one or two orders of magnitude below that concentration.
2. No correlation was found between the turbidity of the dilution water used in chrysotile asbestos dilutions and the resultant asbestos counts.
3. The addition of polyelectrolytes to effect peptization prior to dilution led to more consistent asbestos counts after dilution.

## RECOMMENDATIONS

It is recommended:

(1) that the use of peptization be explored for the preservation of samples stored for asbestos analysis

(2) that the asbestos counting methods be developed to the point where accurate asbestos counts can be made following simple dilution in water.

(3) that a hiatus be instituted in further experimentation on chrysotile asbestiform fibres removal from water until an accurate method of counting is developed.

## References

(1) Hunsinger, R.B., Lawrence, J., and Roberts, K.J. "Pilot Plant Studies to Effect Chrysotile Asbestos Fibre Reduction During Potable Water Treatment" Ontario Ministry of the Environment, Toronto, Ontario, Canada or Canada Centre for Inland Waters, Department of Fisheries and Environment, Canada, Burlington, Ontario.

(2) Lawrence, J., and Zimmermann, H.W., "Asbestos in Water: Mining and Processing Effluent Treatment", Journal of Water Pollution Control Federation, Vol. 49, pg. 156-160 (1977).

(3) Cat-Floc B and Purifloc C-31 are trade names of the Calgon Corporation and Dow Chemical Ltd. respectively.

## ACKNOWLEDGEMENTS

The preliminary results of this project were presented at one of the Ministry of the Environment, Water Technology Section monthly project presentation seminars; Mr. F.J. DART suggested the idea of peptization in order to increase the accuracy of asbestos retrieval after dilution.

## Appendix

### Analytical Techniques for Asbestos Fibres in Water

The sample preparation and counting procedures used for this project are those previously described by Durham and Pang (1). A 250 ml sample was centrifuged at 20,000 rev. $min^{-1}$ (30,000 g) for 2 hours and the separated fibres ultrasonically resuspended in 1 ml distilled water. One micro-litre of this suspension was then transferred to a 3 $mm^2$ carbon coated grid, dried and the fibres counted under a Siemens 101 transmission electron microscope (x 20,000 magnification). When the concentration of fibres was low ($10^5$ - $10^6$ $\ell^{-1}$), it was necessary to count all of the approximately 100 grid openings covered by the 1 $\mu\ell$ drop. At higher concentrations, it was sufficient to count only 20-50 squares and assume that these squares were representative of the entire drop. Background counts were carried out on each grid before use and this number subtracted from the total number of fibres counted for the sample. The limit of detectability was 5 x $10^4$ fibres $\ell^{-1}$. For further details, the reader should consult the original reference by Durham & Pang (1).

### Specimen Calculation

Experimental observation - 60 fibres in 50 squares
Background count - 15 fibres in 100 squares

This is equivalent to 120 fibres in 100 squares (approximate coverage of a 1 $\mu\ell$ drop).

Less background = 105 fibres in 100 squares

Since fibres cannot be observed on the grid bars, a factor of 2.36 is introduced (derived from calculation of the ratio of holes in the grid to the total area).

1 μℓ of concentrated suspension contains

$$105 \times 2.36 = 248 \text{ fibres}$$

1 ml of suspension (all fibres from 250 ml of sample) contains

$$248 \times 10^3 \text{ fibres}$$

1 litre of original sample contained

$$248 \times 10^3 \times 4 = 9.92 \times 10^5 \text{ fibres}$$

Sample preparation techniques do differ from one laboratory to another (eg. centrifuging, filtering with subsequent ashing of the filter media or filtering and removing the media with organic solvent in a condensation washer) and at the present time, no one technique has been accepted as the standard. At the time of commencing this project, Durham and Pang [1] claimed the precision of the analytical method was about $\pm$ 15% but more recent studies and interlaboratory comparisons have necessitated a re-assessment of this value. In 1975, the Ontario Ministry of the Environment arranged for several split samples to be analysed at three independent Ontario Laboratories; discrepancies for any given sample varied by up to two orders of magnitude with complete disagreement on the type of asbestos fibres present [2]. An Asbestos Methodology Committee, sponsored by the Ontario Ministry of the Environment, is currently evaluating and assessing the various analytical techniques and hopefully a standard procedure will be formulated in the near future. In view of the recent work by the Ontario Research Foundation [3], it was felt that the best analytical results obtained during the current project contained an overall uncertainty of at least a factor of 2 (i.e. a sample reported at $1 \times 10^6$ fibres $\ell^{-1}$ could have values from $5 \times 10^5$ fibres $\ell^{-1}$ to $2 \times 10^6$ fibres $\ell^{-1}$).

## References

(1) Durham, R.W., and Pang, T.W.S., "Asbestos Fibres in Lake Superior", Amer. Soc. Testing Materials, Special Publication. No. 573, pp. 5-13 (1975).

(2) Ontario Ministry of the Environment, "An Inter-laboratory Study of Asbestiform Mineral Fibre Levels in the Water Supply of Thunder Bay, Ontario". September 1975 .

(3) Ontario Research Foundation, "Improved Methodology for Determination of Asbestos as a Water Pollutant", Performed under contract for Canada Centre for Inland Waters, Burlington, Ontario (1976).

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